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DAIRY BRANCH.

Mitchell-Walker Moisture Test

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In October we began work on the problem of rapid moisture determination in dairy products. Owing to the increasing demand, of late years, for a practical moisture test to be used in creameries and cheese factories, not a few methods have appeared attempting to meet this demand. We examined the reports of others upon the various moisture tests that are on the market, and in addition fully investigated a number of them ourselves. We concluded that the only feasible methods were the beaker method and Gray's test. The beaker method consists in weighing accurately into an aluminum cup a small quantity of butter, heating it over an alcohol flame till the moisture is all driven off, allowing to cool and reweighing. The loss in weight represents the moisture. From this the percentage of moisture in the sample is calculated. We found some undesirable features connected with this method, viz.,

(1) The necessity of having a very sensitive balance with attached weights, or else having a cheaper balance with separate weights which are rather tedious to use and liable to get lost in practical work in factories.

(2) The necessity of calculating from the weights obtained the percentage of moisture in the sample.

(3) The uncertainty as to the right moment to stop the heating.

(4) The care required to prevent loss by foaming or spattering.

(5) The uncertainty as to the right time to reweigh after heating, since if reweighed when still warm a wrong result is obtained, and if allowed to stand too long in the open air before reweighing the dried sample absorbs moisture from the air, and hence again a wrong result is obtained.

(6) The necessity of weighing accurately to the second decimal place, in grams, to give a sufficiently accurate result.

Upon the whole we concluded that this method which has been considered by several chemists of note as the best rapid test yet offered, although quite reliable in the hands of a careful worker in a chemical laboratory, is not suitable in the hands of the average man. Not to our knowledge has it ever been used or recommended for work with curd. We tested it with curd and found it to work fairly well, but of course the above-mentioned objections, with the exception of number 4, still hold here. Several modifications of the beaker method have appeared from time to time.

Gray's test, consisting of heating a ten gram sample of butter with a mixture of amyl acetate and amyl valerianate in a glass flask, to which are attached a glass condenser and graduated receiver reading in percentages, presented none of the above difficulties; but we found several objections to it of a much more serious nature. It gave, with us, readings altogether false as compared with the official method, concerning which there is no doubt whatever as to its reliability. Further, so much care is required to prevent loss of moisture through the open graduated neck and to guard against the breaking of a comparatively expensive and fragile piece of glassware, that these points themselves would tend to militate against its use in a creamery. In addition, the apparatus requires putting the different portions together and taking them apart again for each test. Lastly, the reagent used is rather expensive for practical use, since it cannot be used over again.

Since Gray's form of apparatus appeared on the market a so-called "improved form" of it has been brought forward. We tested this, also, but found it more fragile, rather cumbersome, and requiring considerable time and trouble to clean, although it gave more accurate readings than the original form. This greater degree of accuracy is due to a modified form of graduation of the scale. The errors in the results with the original form of Gray's test are due, chiefly, to absorption of moisture by the reagent used and not to any lack of condensation, providing sufficient care is taken in conducting the test.

Being dissatisfied with all the methods investigated by us, we undertook [illegible] a form of moisture test that would prove more satisfactory [illegible] decided that the following requisites were necessary, and we kept [illegible] continually in view during our work:

(1) A rapid and reasonably accurate method.

(2) An inexpensive form of apparatus, both to purchase and to use.

(3) A durable form of apparatus and one easy to clean.

(4) A method that requires no great amount of attention or care in operating, and that can thus be used by the average factory man.

(5) An apparatus than can, if damaged, be easily repaired at small cost.

(6) A method equally suitable for testing butter, curd and cheese.

The next thing to decide on was, of course, the underlying principle to make use of. The only one that appeared to warant development was that of using a reagent, expelling the moisture with heat, condensing the vapor and collecting it in a vessel graduated to read directly in percentages.

The next points to settle were the best form of apparatus and the best inexpensive reagent to use.

At the outset we determined to use metal in the apparatus, so far as possible, rather than glass, owing to the danger of breaking the latter, and to use no rubber connections. We thought to make the condenser and heating flask of aluminum, if possible, owing to its many desirable qualities. As no satisfactory solder for aluminum has, as yet, appeared we could not attach an evaporating cup to the condenser, unless we used some form of shoulder-and-nut connection, which did not seem altogether desirable, nor could we attach an inner condenser tube to an outer water jacket unless we used rubber stoppers at the ends. Further, upon experimenting to determine the effect, if any, of reagents upon sheet aluminum, we found that the aluminum became roughened too much to warrant its use as an inner condenser tube.

We then tested the effect of reagents on other metals, finding that block tin showed no ill effect whatever. We decided then to use a block tin tube for the inner condenser tube. This was sufficiently cheap, and could be easily soldered to the outer water jacket and to the evaporating cup. We had the outer water jacket made of thin sheet brass. The best size of tin tubing to use had to be determined by experiment and also the size of the water jacket.

We decided that to insure complete condensation of the water, without making the condenser too cumbersome, the vapor should be driven downward instead of upward. This would thus insure the lower part of the condenser being cold, since the warmed water would remain at the top. We wished further to have a condenser large enough to hold sufficient water not to need refilling under average conditions in a factory.

The object of using a reagent was to have something to keep the butter from foaming too much during the heating process, to help carry the condensed moisture out of the inner condenser tube, and to wash the tube out thoroughly afterward. We tested the use of amyl butyrate at this stage, since it was almost insoluble in water and was sufficiently volatile for part of it to be driven off with the moisture, and at the same time had a boiling point sufficiently high for part of it to remain after all the moisture was expelled from the sample, and pass over later to serve as a wash for the inner condenser tube. We were unable to procure a suitable metal evaporating cup at first, and in the meantime used a glass flask, connecting it with the condenser by means of a perforated rubber stopper. We used the condenser in a slanting position at first, but found that in this position the inner tube did not become thoroughly washed out by the reagent. We then used it in an upright position. This modification and the use of an inner tube of suitable bore, formed a combination that worked admirably, causing the inner tube to flush quickly and thoroughly. It necessitated considerable experimenting to ascertain the best size of inner tube to use, one neither too large nor yet too small in bore, as each extreme presented its own difficulties.

The graduated receiving vessel had, of course, to be made of glass. We tried several forms and sizes, finally adopting that shown (and marked C.) in the illustration appearing in the bulletin. The oval-shaped, wide-mouthed upper portion forms a suitable receiver for the condensed moisture and reagent and a reservoir for the latter, while the constriction and outward curvature at the mouth and the spring wire clamp on the bottom of the condenser form a combination for readily attaching the receiver to the condenser and just as readily detaching it. The tube forming the lower portion of the receiver is of suitable bore to be graduated for reading per cent. of moisture for the size of sample taken, while the stop-cock at the bottom is for emptying purposes.

We wished to have the whole apparatus so constructed that it could be readily taken apart and packed for use by travelling inspectors, and in this we feel that we have been singularly successful.

For the purposes of this test, we found that a sufficiently accurate, and otherwise suitable, balance was obtainable at a reasonable price, thus making the whole apparatus really no more expensive than would be a balance alone which would be sufficiently accurate to be used in connection with any of the beaker tests, since they necessitate the use of a balance that will weigh accurately, in grams, to the second decimal place. To facilitate the work of readily balancing the cup on it for weighing the sample we devised a counterpoise to be attached to one of the arms of the balance. (See illustration.) This saves time in the weighing of the sample, making it a very quick process.

We felt that the only problems left to solve were those of getting a suitable metal evaporating cup, and of satisfying ourselves with regard to the reagent to [illegible] After some difficulty we succeeded in getting a cup of thin sheet [illegible]per made by the spinning process, thus eliminating the use of solder, which would melt under the heat applied. We had considerable difficulty in getting a satisfactory steam-tight joint in the cup, however, but we have proved that the one we are now using is perfectly reliable. The form of cup is cylindrical, which renders it easy to weigh the sample into and to clean.

The problem of the best and cheapest reagent to use proved to be the hardest of all to solve. The main difficulty, outside of the feature of expense, lay in the fact that all the reagents we tried (and we tried many) seemed to absorb part of the moisture driven from the sample under the test, and thus delivered a shortage of water, and caused the readings to be too low. We had the receiving tube graduated to read in percentages, providing a ten gram sample were used and all the moisture were recovered, that is, a volume of water recovered measuring one cubic centimetre would read ten per cent. on the scale of our receiver. After obtaining no reagent that would deliver all the water we concluded that we would have to just decide upon some one reagent, and compare the results from its use with those obtained from the official method; that is, heating a very accurately weighed sample in a water oven to constant weight, reweighing and calculating the percentage of moisture from the loss in weight from the heating process. Now, amyl butyrate seemed to be a fairly desirable reagent, although it had some undesirable features, namely, that it was rather expensive—especially the chemically pure product; its boiling point was rather high, requiring a longer time than we thought desirable to drive enough of it over to wash out the inner tube of the condenser, and its odor, although pleasant under ordinary circumstances, proved to be disagreeable when heated with butter, or when in contact for any length of time with the hands. We tried a large number of reagents, among which were eucalyptol, amyl acetate and various distillation products of crude petroleum. In choosing a reagent we had to consider the various requisites of one suitable for our purpose, namely, its boiling point, specific gravity, ability to prevent foaming of the sample, stability under the conditions to which it would be subjected, its suitability for flushing the inner tube of the condenser and finally its cost. Although we could easily have chosen a reagent consisting of a mixture of substances which would have performed the work well enough, yet, as we were anxious to use our reagent over and over again to reduce the expense of testing, we had to discard the idea of a mixture, since the constituents of highest boiling points would partly remain behind in the evaporating cup, thus altering the composition of that portion of the reagent recovered. We finally concluded that amyl acetate, when chemically pure, fulfilled these requisites in the largest measure.

After thus getting our method approximately complete, we concentrated our efforts upon solving the problem connected with its accuracy. We first used it with butter, and after making an exhaustive number of tests with this product we discovered that the *loss of moisture due to the reagent approximated a constant quantity, rather than being proportional to the percentage of the moisture in the samples.* We next used our test with curd and cheese and found the same principle to hold here also. The explanation of this appears to us to be that the moisture lost is that absorbed by the portion of the reagent that volatilizes with the moisture, the balance of the reagent being recovered in a moisture-free condition.

We thus found it necessary to correct the graduations on the receiving tube accordingly, so that it now gives readings closely approximating the results obtained by the official method, as may be seen from an examination of the table of results obtained by us.

RELIABILITY OF THE TEST.

That the test is reliable and more than approximately accurate is evident from the results recorded in the following tables :

Butter Samples.	Gravimetric Method.	Rapid Method.
1	11.62	11.7 11.8
2	15 31	15.3 15.4
3	12.38	12.4 12.5
4	9.90	9.8 9.8
5	14.50	14.4 14.4
6	10.83	10.95 10.95
7	11.64	11.65 11.7
8	19.53	19.6 19.6
9	16.77	16.7 16.7
10	12.10	12.2 12.2
11	12.08	12.0 12.1
12	14.0	14.1 14.1

Curd Samples.	Gravimetric Method.	Rapid Method.
1	39.6	39.6 39.6
2	55.38	55.4 55.4
3	53.12	53.2 53.0
4	49.07	49.2 49.4
5	61.83	61.6 62.0
Cheese Samples.		
1	30.53	30.4 30.2
2	35.0	34.8 34.6

RAPIDITY OF THE TEST.

Our methods of preparing the samples are very simple, reliable and rapid, while the thin, metal evaporating cup and the style of condenser used permit of rapid evaporation and insure condensation of the moisture without danger of either breakages or loss of moisture.

The time required for driving all the moisture from a sample of butter or curd is from four to seven minutes.

TEST SIMPLE TO CONDUCT AND APPARATUS EASY TO CLEAN.

An examination of the cut and a reading of the description of the test and the directions for conducting it will satisfy the reader as to its simplicity. The apparatus is simple in all its parts, while a single reagent, *not a mixture,* is used in the test.

There are very few pieces to clean and these are so constructed as to facilitate the work of cleaning them. The size and shape (cylindrical) of the evaporating cup render it very easy, both to fill and to clean, while the reagent prevents the sample from adhering to it. All other parts are just as easily cleaned.

Small Cost of Conducting a Test.

The chief item of expense, in conducting a test, is the reagent used. This we have succeeded in reducing to a minimum through making provision for using the reagent over and over again. The average cost per test for both butter and cheese is somewhat below half a cent.

Durability of the Apparatus and Cost of Repairs.

Practically the whole of the apparatus is made of metal—the evaporating cup, the condenser and the spirit lamp—and, in addition, it is made in sections. It is thus seen that the apparatus is durable, the chances for breakages are slight and repairs can be made at a trifling cost. Everything considered, the apparatus is an economical one both to purchase and to use.

Other Uses for the Test.

While originally designed for use in connection with cheese and butter making, the test will doubtless serve a much wider field than that for which it was devised. We have used the apparatus for determining the percentage of moisture in flour, bread, breakfast foods, wood pulp, etc., with good results.

EXPLANATION AND DESCRIPTION OF THE TEST AND APPARATUS, AS SHOWN IN THE ACCOMPANYING ILLUSTRATION.

Explanation of the Test.

A representative sample of the substance to be tested having been obtained, the required quantity of it, 10 grams of butter or 5 grams of curd or cheese, is weighed into the evaporating cup A. To this is added the reagent. After connecting the cup with its cover the moisture and reagent are evaporated by means of the spirit lamp E. The vapors are condensed to liquid form again in passing through the small tube b2, of the condenser B—this small tube being surrounded by cold water—and the condensed moisture and reagent flow out of the condenser into the graduated glass receiver C. As the water and the reagent do not mix, the water, which is the heavier, settles to the bottom of the graduated tubular neck of the receiver.

The scales on this neck are graduated for reading, directly, the per cent. of moisture in a sample when either 5 or 10 grams are taken to a test.

An examination of the accompanying cut shows the whole apparatus—lamp, cup, condenser and receiver—to be supported, when in use, by the upright K, which screws into a plate on the top of the base H. Once the sample is weighed and the heat applied the work goes on almost or quite automatically; and as there is no delicate glassware that requires close attention and no danger of the escape and loss of any of the moisture, the operator is at liberty, during the short time that it takes to evaporate the moisture, to attend to other small duties, if circumstances call for it, as they frequently do.

The reagent used serves several purposes. It prevents foaming and keeps the sample from adhering to the bottom of the cup or charring unduly; a portion of it passes over with the moisture and aids in carrying it both out of the condenser and down the receiver; while the high boiling point of the reagent causes a portion of it to remain behind in the cup, pass over later and thoroughly wash all the moisture out of the small tube of the condenser. This last is very much facilitated by the upright position of the condenser.

Description of the Apparatus.

A. Evaporating cup (metal).
  a1. Evaporating cup proper.
  a2. Lid of cup.
B. Condenser.
  b1. Jacket of condenser for holding water.
  b2. Block tin condenser tube through which the vapors pass to be condensed.
  b3. Spring clamp for holding the graduated receiver, C.
C. Graduated glass receiver. The condensed liquids flow into this, the water settling to the bottom of the graduated tubular neck. The receiver has two scales, one for reading per cent. for 5 grams and the other for reading for 10 grams.
D. *Balance* for weighing the sample. The cup, A, is placed in one pan and is readily balanced by means of the counterpoise on the opposite beam. The weight is then placed in the other pan for weighing the sample to be tested.
E. *Spirit lamp* (metal for evaporating the moisture from the sample.
F. Reagent outfit.
  f1. Bottle of reagent prepared for use.
  f2. Bottle for receiving used reagent.
  f3. Graduate for measuring reagent.
G. Alcohol bottle for holding wood alcohol to be used in the spirit lamp.
H. Stand or base for apparatus.
K. Upright which screws into the base H.

L. Sampling cup and spoon for preparing sample of butter and transferring it to evaporating cup on balance.

M. Curd and cheese sampler.
  - m1. Cylinder for taking sample. It has a wire gauze on one end.
  - m2. Piston for forcing the sample through sieve or gauze on the end of m1, thus cutting it into fine particles.

DIRECTIONS FOR CONDUCTING TEST.

To Test Butter.

1. Set up the apparatus just as it appears in the cut.

2. Fill the condenser with cold water.

3. Obtain a representative sample of butter and place it in the sampling cup, L. Heat *carefully*, either in hot water or over the alcohol flame, stirring continuously with the spoon provided until the butter is all *melted to a creamy consistency.*

4. Place the evaporating cup, A, on the scale pan and balance it by means of the counterpoise on the opposite beam. Place the *10 gram* weight on the other scale pan and after again stirring the sample thoroughly, transfer sufficient of it to the cup to exactly balance the 10 gram weight.

5. Using the graduate, f3, add 10 cubic centimeters (c.c.) of the reagent to the cup from the bottle f1.

6. Connect the cup with its lid a2.

7. Apply the alcohol flame (a fairly large flame) to the evaporating cup—the lamp can be readily adjusted by moving it on the upright. In about one minute the water and reagent will begin to pass over and drop from the condenser tube, b2, into the receiver, C. After all the water has been evaporated from the cup the reagent will cease, or almost cease, dropping for a moment and then begin again as soon as it has reached its own boiling point, which is higher than that of water. Continue to apply the flame until practically all the reagent is driven off and it ceases to drop freely from the condenser tube. By this means all the water is washed out of the condenser tube and the major portion of the reagent is recovered. Now swing the alcohol lamp to one side and *put its lid on.*

8. Immediately remove the receiver, C, cork its mouth and, taking it by the top, swing it carefully a few times to detach any drops of water that may be adhering to its sides. Read the percentage of moisture direct from the scale, taking the bottom of the meniscus between the water and reagent as the top of the water line. Should any particles of moisture still adhere to the side of the tube, or the meniscus be imperfect, the wire accompanying the apparatus can be used to remedy this.

9. Have two reagent bottles and label them 1 and 2.
   No. 1 contains the reagent ready for use.
   No. 2 is for the used reagent.

10. When emptying the receiver, C, first open the stopcock to allow all the water to run out, then close it and then hold the receiver over bottle No. 2 and allow the used reagent to run into it. The receiver, after being shaken, is ready for use again.

11. Bottle No. 2 is necessary for keeping the used reagent separate from the other, since it has absorbed a small amount of moisture from

which it must be freed before it can be used again, else it would give too high a reading. When the water-free reagent in bottle No. 1 has been all, or nearly all, used up, the used reagent in bottle No. 2 can be freed of moisture by filling the carefully cleaned evaporating cup with it, attaching it to the condenser and heating *until water ceases to drop* from the condenser tube. The drops of water are readily seen as they sink through the reagent in the receiver. Now remove the cup and empty its contents, which are free of moisture and ready for use, into bottle No. 1. Put the reagent in the receiver into bottle No. 2, as it contains moisture.

CAUTIONS.

1. Always empty the reagent in the receiver, C, into bottle No. 2.
2. Never use the reagent from bottle No. 2 for testing; use only that from bottle No. 1.

TO TEST CURD AND CHEESE.

The directions for testing curd and cheese are similar to those for butter with the exception of the following: The sample is taken by pressing the cylinder, m1, into the curd in the vat or into the cheese, as the case may be, twisting it round and pulling it out. The sample will be found in the cylinder. Be sure to obtain as representative a sample as possible. The piston, m2, is now pressed in, thus forcing the sample through the sieve on end of cylinder, m1. The sample should be pressed through the sieve once more to reduce it to a fine state of division. Stir the sample with the spoon and transfer it to the evaporating cup on the balance, *using five grams to a test.*

Use fifteen cubic centimeters of the reagent for curd and cheese and add a small piece of paraffin wax to the cup to prevent the sample from adhering to the bottom. After the test the curd or cheese is readily removed by means of the spoon whose upper end is specially fitted for this purpose.

No great care need be taken in cleaning the cup after a test, since it contains no moisture and can be easily balanced on the scale pan by means of the counterpoise. Simply wipe it out with a dry cloth.

DIRECTIONS FOR CLEANING THE APPARATUS AND FILLING CONDENSER.

The condenser need never be refilled with water unless several tests are made in close order. In this case empty the condenser when it becomes very warm about half way down and refill it with cold water.

The inner condenser tube must be cleaned after every few tests, with the flexible brush supplied. This should be run through the tube, first in the dry condition and secondly moistened with the reagent. *Use no water in this operation.*

The receiver, C, may be cleaned in a similar manner, although it is advisable to *occasionally* clean it with a hot solution of washing soda, followed by alcohol and this again by a little of the reagent.

We could have made our directions very brief had it seemed advisable to do so, but we preferred making them sufficiently full and explicit to cover and make clear all points in connection with the test, and thus insure accurate results. This course will, we believe, be appreciated by users of the test.

After making a few tests the simplicity of the whole operation will be apparent.

ADDENDA.

Experience has shown us that the balance works better with the heavy part of the counterpoise below instead of above the beam, and we have altered it accordingly. Place the counterpoise on the beam so that it practically balances the cup. Finer adjustments after this has been done are made by means of the nut on the screw that projects beyond the beam.

Keep the glass stopper of the graduated receiver vaselined.

Experience with both a threaded and a friction joint on the cup has proven that the latter works the better.

When testing curd or cheese heat more slowly than with butter, taking from seven to eight minutes to drive the moisture off.

The reagent used in this test is amyl acetate (technical variety), and should be obtainable from any reliable druggist at about one dollar per pound. It should be put in the cup and tested for moisture, and, if necessary, freed from the same before using.

PRICE OF THE MOISTURE TEST.

The apparatus may be obtained from us at the following prices: complete outfit, including an 8 oz. bottle of reagent, $13. Travelling case for outfit, $2 additional. (This is furnished only when specially ordered.) In cases where the balance for weighing out samples is not wished, a deduction of $3.50 from the above price is made. We recommend the use of the balance supplied, however, as it has a counterpoise to balance the cup, and is otherwise specially fitted for the work.

Individual parts may be obtained at any time at reasonable prices. Customers in Canada will be charged $14 for the complete outfit, as parts of it have to be imported and duty paid thereon.

All orders, unless accompanied by the price, sent C.O.D.

Send all orders for apparatus to Supt. of Eastern Dairy School, Kingston, Ontario.